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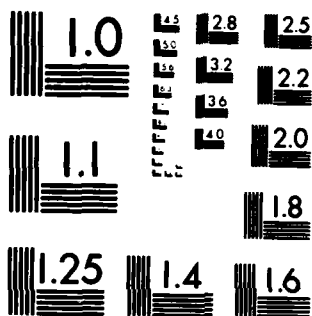   AERONAUTICAL LABS WRIGHT-PATTERSON AFB OH   P L BUCKLEY

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A STUDY OF THE USE OF TRANSPARENT YTTRIUM OXIDE FOR RAMJET COMBUSTION RESEARCH

Parker L. Buckley

August 1982

Final Report for Period January 1981 to April 1981
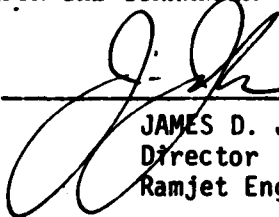
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Yttrium oxide is considered as a candidate material for the fabrication of a transparent ramjet combustor to be used for combustion research. Data from several sources is compiled to compare various processing techniques. Information is provided on yttria powder preparation, sintering techniques, and on variations such as hot forging and the use of dopants to achieve theoretical density. Mechanical properties are discussed as related to the combustor application.

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FOREWORD

This report was prepared by Parker L. Buckley of the Ramjet Technology Branch, Ramjet Engine Division, Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories (AFWAL/PORT), Wright-Patterson Air Force Base, Ohio 45433. The work was accomplished under Work Unit 2308S101 "Ramjet Combustors."

The report covers research performed during the period January 1981 to April 1981.

This report was submitted for publication by the author in March 1982.

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SECTION I

INTRODUCTION

Recent in-house experimental work in the Ramjet Division of the Aero Propulsion Laboratory at Wright-Patterson Air Force Base has been directed toward the characterization of the flowfield inside the ramjet combustor. The ramjet configuration of primary interest is the simple coaxial dump combustor in which the inlet air enters the combustor in the forward end by a sudden expansion about the centerline. The result is a very complex reacting flow, including recirculation and possible combustion instabilities. The problem is further complicated when swirl addition or the use of flameholders is considered (Reference 1).

Direct measurement of properties inside the combustor is very difficult. Hardware such as directional pitot probes and thermocouples must be able to withstand a high velocity oxidizing atmosphere with pressures of up to 100 psia and temperatures up to 2200°C. Also, measurements taken with probes large enough to be water-cooled are suspect due to errors introduced by the presence of the probe. Thus, an attractive alternative is optical measurement.

Several different approaches to visual observation or optical measurement have been tried. One of the most basic of these has been the use of a water tunnel constructed of clear plexiglass in the same configuration as the actual ramjet hardware. Water is pumped through the model at a rate sufficient to provide an inlet Reynolds number equivalent to that of the ramjet. Dye or bubbles may be injected to trace the flow path through the system. Results are obtained by simple visual observation or by the use of high-speed motion pictures. A logical extension of the technique is to try to construct a transparent ramjet combustor in which the actual combustion process can be photographed. Thus, hardware was designed and fabricated which incorporated a six inch I. D., 1/4 inch wall quartz tube, 24 inches long, as the ramjet combustor. Several tests have now been made using this transparent combustor with varying degrees of success. If the operating conditions are closely controlled, test times of several seconds are possible with very good

photographic results. However, operating the ramjet with combustion
instabilities or for excessive run times has resulted in bursting the
quartz tube.

Recent in-house work with a laser doppler velocimeter (LDV) system
has progressed to the point of attempting to use this equipment to
optically determine velocity components inside the ramjet during combus-
tion. This system incorporates an argon-ion laser (green) which is used
to form an interference grid at the intersection of two or more laser beams.
The properties of this grid are known with sufficient detail that parti-
cles, seeded into the airflow, may be timed as they pass through this
area, allowing determination of the velocity components. This small
test volume can be traversed across and along the combustor to develop a
complete flowfield analysis. Problems arise, however, when attempting
to use the LDV system in conjunction with the quartz chamber. Of primary
concern is damage to the equipment. While a high-speed movie camera can
be used at a distance of several feet from the combustor and can be pro-
vided with a protective housing, the LDV system must be operated within
a few inches of the combustor with very little protection. Thus, it
becomes very desirable to develop a more durable transparent combustor.

One interesting material to consider for this application is yttrium
oxide, or yttria. Through the use of suitable sintering techniques, this
ceramic material can be made transparent to visible light while maintain-
ing excellent high temperature properties. The following report serves
to compile information about the fabrication and mechanical properties of
yttrium oxide with the idea of using this material in ramjet combustion
research.

SECTION II

FORMATION AND PROCESSING

Yttrium oxide, Y_2O_3, is a naturally occurring compound, first discovered in 1788 in a quarry near Ytterby, Sweden. It was not until 1843 that relatively pure yttrium oxide was extracted from this new mineral "Ytterbite." By the mid-nineteenth century yttrium was determined to be trivalent with an atomic weight of 89. Today, yttria may be obtained by the ignition of yttrium compounds containing organic components or volatile oxy-acids (Reference 2).

The yttria crystal is of cubic configuration with a lattice constant of 10.605A°. The distance between the yttrium and oxygen atoms is 2.27A°. Although cubic, the yttria crystal structure is quite complex as shown in the figure below (extracted from Reference 3).
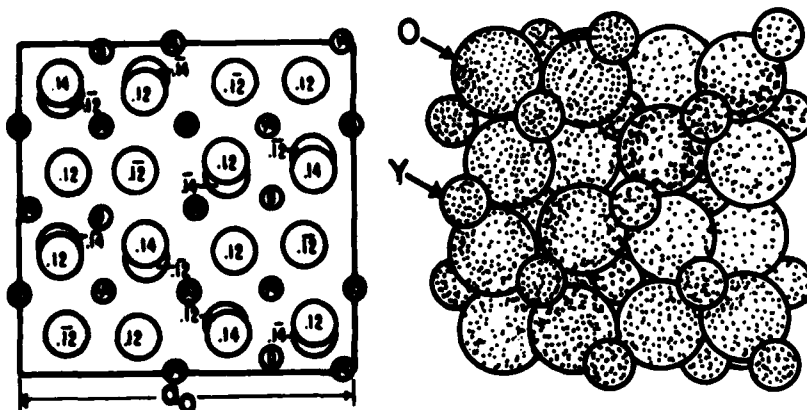
With suitable processing, yttrium oxide ceramics can be made transparent to visible light. This occurs as a result of energy in the visible light spectrum not being absorbed by free electrons within the crystal. If electrons were free to vibrate at their natural frequency as light passed through the crystal, that portion of the spectrum corresponding to the frequency of the electron vibration would be absorbed (Reference 4). If a band of electron vibrations spanned the entire visible spectrum, the material would be rendered opaque. Almost all of the available lit-

erature, however, is limited to empirically derived procedures for obtaining tansparent yttrium oxide, rather than dealing with internal electron activity.

1. FABRICATION TECHNIQUES

The fabrication of useable objects or test specimens from yttrium oxide is primarily a sintering operation in which suitably prepared yttria powder is pressed into shape and subsequently or simultaneously heated to a temperature below the melting point. During this time at elevated temperature, bonding of the powder results as vacancies diffuse from pores to grain boundaries formed by the original particles. As the vacancies are eliminated, shrinkage of the material occurs. If the diffusion of vacancies from the pores to the grain boundaries is sufficient, it is possible to achieve the theoretical density of the yttria (Reference 5). Variations include hot forging under high pressure (Reference 6) and adding dopants such as thoria to control grain growth during sintering (Reference 7).

a. Powder Preparation

A very complete description of two methods of powder preparation is given in Reference 7. The first of these, the sulfate process, consists of mixing an aqueous suspension of yttrium oxide powder with an aqueous solution of thorium and neodymium sulfates. The total composition was tailored to result in 89 mole % Y_2O_3, 10 mole % ThO_2 to inhibit grain growth, and 1 mole % Nd_2O_3 used for subsequent lasing measurements. Mixing was accomplished by colloid milling, then magnetic stirring with the simultaneous application of heat to dry the suspension. After extended drying in 110°C air and screening, the sulfates were converted to oxides by calcinating for seven hours in 1000°C air. Finally, the powder was pressed into one inch disks, 3/16 inch thick or into 3/10 inch diameter rods, 4-1/2 inches long, then sintered at 2170°C.

In the second technique, the oxalate process, the same composition of Y_2O_3, ThO_2, and Nd_2O_3 was desired. Appropriate portions of yttrium, thorium, and neodymium nitrates were dissolved in water, then dripped into an oxalic acid bath, resulting in an oxalic precipitate. After

washing to remove excess acid, drying, etc., the oxalate was calcinated
in air for four hours at 800 - 850°C. The calcined powder was pressed
into shape or milled further before processing, then sintered as before.

Both processes result in high optical quality yttrium oxide with the
milles oxalate powder process yielding a residual porosity of less than
one part per million. This process was preferred in other works (Refer-
ences 8, 9) for laser applications due to the increased sintering rates
with smaller particles and reduced heat treatment required to eliminate
sulfur from the sulfate process.

The simplest powder preparation was described in Reference 6 in con-
junction with the hot forging technique. The yttrium oxide powder
(average 3 micron) was milled with 3 to 5 percent lithium fluoride (-325
mesh) for 12 hours in a plastic container with alumina milling cylinders
ar.d acetone as the milling vehicle. After milling, the acetone was
evaporated, then the powder was dried at 60°C in a vacuum for 12 to 24
hours. Finally, the powder was cold pressed at 3000 psi into 7/8-inch
diameter disks in preparation for the hot forging process.

b. Sintering and the Control of Grain Size

An excellent sequence of photographs published by J. E. Burke
(Reference 10) provides insight into the elimination of pores during
sintering. In the first of these, alumina powder is shown as sintered
for one hour at 1800°C. The average grain measures about 15 microns,
which is close to the original particle size in the powder. Numerous
pores are to be seen located on the grain boundaries due to mismatches
in the original single crystal particles. After additional heating for
one hour at 1900°C, extensive grain growth has occurred with an average
grain size now greater than 300 microns, but the original pores have
remained. Further heating for two hours at 1950°C tends to eliminate
many pores near grain boundaries, but pores located away from boundaries
have grown in size. This process is explained as lattice diffusion
whereby vacancies diffuse from pores to nearby grain boundaries, while
those located more distant from boundaries act as vacancy sinks and grow
in size as smaller pores are eliminated.

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The mechanism of grain growth is described by Burke as a migration of grain boundaries toward their centers of curvature. During this process, some grains will disappear as others grow larger, but in general, the grain size will remain fairly uniforn at any given time. This process may be halted when inclusions or pores restrict the grain boundary motion. At the same time, however, an exaggerated or "discontinuous" grain growth can occur when the disappearance of pores allows a few grains to grow and gain more sides than their neighbors. The growth force of these grains, being proportional to the number of sides (Reference 10), is increased. This triggers an unstable growth of a few grains to dispro-portionate size at the expense of the average sized grains. Many pores are trapped in these larger grains and can only be eliminated by exces-sive sintering times.

Thus, if grain growth can be controlled during sintering, fewer pores become isolated in the center of very large grains; consequently, vacan-cies are more readily diffused into the boundary areas and eliminated. As this occurs, shrinkage to theoretical density is possible.

A great deal of work has been done at General Electric with thorium oxide dopants to control grain growth in yttrium oxide (References 5, 7, 8, 9, 11). The commercial product name for this material is Yttralox. A detailed account of the procedures for incorporating ThO_2, ZrO_2, HfO_2 and combinations of these is also reported in a patent issued to one of the principal investigators at General Electric (Reference 12). In Reference 5, microhardness measurements indicate that as thoria is added to yttria, both the internal grain and grain boundary hardness is increased, but the grain boundary hardness always increases at a higher rate. This data is interpreted as a solution hardening phenomena, while increased boundary hardness is a result of a greater thoria concentration at the grain boundaries. It is interesting to note that previously sintered test samples could be reground and sintered again with the same result. The optimum thoria content was determined to be 7 to 10 mole % to achieve fully dense yttrium oxide. The lattice constant of the yttria is a linear function of thoria content, increasing from 10.605 Å for undoped yttria to 10.65 Å for 10 mole % thoria addition. The end result of thorium

doping is decreased mobility of the grain boundaries with reduced grain growth during sintering.

Lefever and Marks at Sandia Laboratory have produced transparent yttrium oxide disks by a hot forging technique (Reference 6). In this work, the yttria powder (average particle size 3 microns) was milled with 3 to 5 weight percent lithium flouride. After milling, (described previously) the powder was sintered under vacuum at 950°C while a mechanical pressure of 10,000 to 12,000 psi was applied. Transparent disks one centimeter in diameter and several millimeters thick were produced in this manner. The lithium addition was claimed to be required for full transparency, but the exact mechanism was not reported. Flame photometry indicated that most of the lithium was eliminated during the sintering process. The average grain size was measured as three microns; thus, grain growth did not occur. The density of the yttria was determined to be 5.030 g/cc compared to the theoretical value of 5.031 g/cc as determined by X-ray techniques. Samples were also produced with a europium oxide dopant of .5 mole percent. Little data was presented, but the Eu_2O_3 apparently remains in the yttria, much like the thoria dopant used by General Electric. Light transmission data was very similar to the non-doped sample.

In a patent issued to the United States Army (Reference 13), a method is described for producing transparent yttria by vacuum hot pressing of cold pressed powder compacts in a graphite die. Advantages claimed for this technique include a grain size of less than one micron, improved tensile strength, elimination of additives or dopants, and reduced processing time. The procedure reproduced here is taken from the patent description.

    a. Selecting a yttrium oxide powder having a purity 99.90 - 99.99%.

    b. Cold pressing the powder to form a compact 45 - 65% of theoretical density.

    c. Evacuating the compact at a temperature of 300 - 600°C.

    d. Applying a pressure of 5000 - 7000 psi.

    e. Raising the temperature to 1300 - 1500°C.

f. Maintaining the pressure and temperature for a
    period of 1 - 2 hours, slowly cooling, and re-
    covering transparent yttrium oxide having an
    ultra-fine grain size of less than one micron.

Suggested applications for this material include high intensity lamps,
infrared windows for heat seeking rockets, high temperature windows, and
development of transparent armour for ground vehicles. Material produced
by this technique is claimed to have optical transmittance equal to that
produced by Lefever, et al (Reference 6) and Anderson (References 11, 12).
Increased tensile strength is also claimed, but specific data is not
provided in the patent report.

Most of the yttria products described thus far have been test samples
such as small disks or rods which could be readily produced by the pres-
sing or hot forging techniques. The ability to apply these techniques to
more complicated geometric shapes was not addressed. In Reference 14
(another patent report) an entirely different sintering process is descri-
bed in which .1 to 5.0 weight % magnesia (MgO) is added to the yttria.
The result is liquid phase sintering as opposed to the single phase process
involved in the hot forging or doping technique. One of the prime objec-
tives of the invention is the ability to form transparent yttria bodies
in the form of tubes and other intricate shapes. Powder preparation is
similar to that for the other techniques, but is then cold pressed into
shape at 15,000 to 50,000 psi. Sintering is accomplished for 1 to 6 hours
at temperatures between 1900° and 2200°C. The ability to sinter at
ambient pressures greatly facilitates the formation of more complex
structures.

Another alternative, found in the Soviet literature (Reference 15),
deals with casting yttrium oxide into very complex shapes with a paraffin
wax binder. These objects were actually sintered at 1600 to 1800°C
rather than being liquid cast as the title implies. Also, it appears
that yttrium oxide was chosen for a specific application because of its
high temperature dielectric properties. Transparency was not discussed,
but a theoretical density of up to 99.5% was attained. Thus, wax binding
techniques are probably worth considering for future work with more
intricate shapes.

Numerous other reports are available in foreign literature, especially that of the Soviet Union and Japan. References cited in this report include those most accessible from this country and the few foreign reports for which a translation exists.

In this section, details for various methods of powder preparation, sintering, and controlling such things as grain growth, porosity, and density have been described. The final result, in most cases, was a yttrium oxide sample with good transparency to visible light and 97 to 99.9% theoretical density. In the following section, the data is presented for the physical properties of the yttria.

SECTION III

PHYSICAL PROPERTIES

In this section, the properties of yttrium oxide are discussed and correlated to the appropriate processing techniques where possible. The primary concerns are optical qualities, performance at elevated temperature, and the ability to actually fabricate a transparent ramjet combustor.

1. OPTICAL PROPERTIES

The optical requirements for the application of yttria to the transparent combustor are not particularly stringent. It is important that the material pass a high percentage of visible light, especially the green associated with the argon-ion laser. Plotted in Figure 1 are transmission data from the General Electric Yttralox (Reference 7) and Lefever's (Reference 6) hot forged samples, normalized to 1.0 mm thickness. Yttria produced by the Army's hot pressing technique (Reference 13) is claimed to be comparable. As seen in Figure 1, there is little absorption of visible light (4000 to 7000 Å). Green light extends from approximately 5000 to 5500 Å.

Also important to the optical qualities is the lack of a second phase in the yttria whose refractive index is different from that of the ceramic (Reference 11). The index of refraction for Yttralox is plotted in Reference 11 as a function of wavelength. For green light, it is approximately 1.93. From this data, yttrium oxide appears to have optical qualities quite sufficient for the application considered here.

2. MECHANICAL PROPERTIES

Throughout the section on fabrication techniques, achieving the theoretical density of yttria was of concern in obtaining adequate mechanical and optical qualities. One effect of porosity is illustrated in Figure 2 in a plot of Young's modulus as a function of volume fraction of pores (References 16, 17, 18).
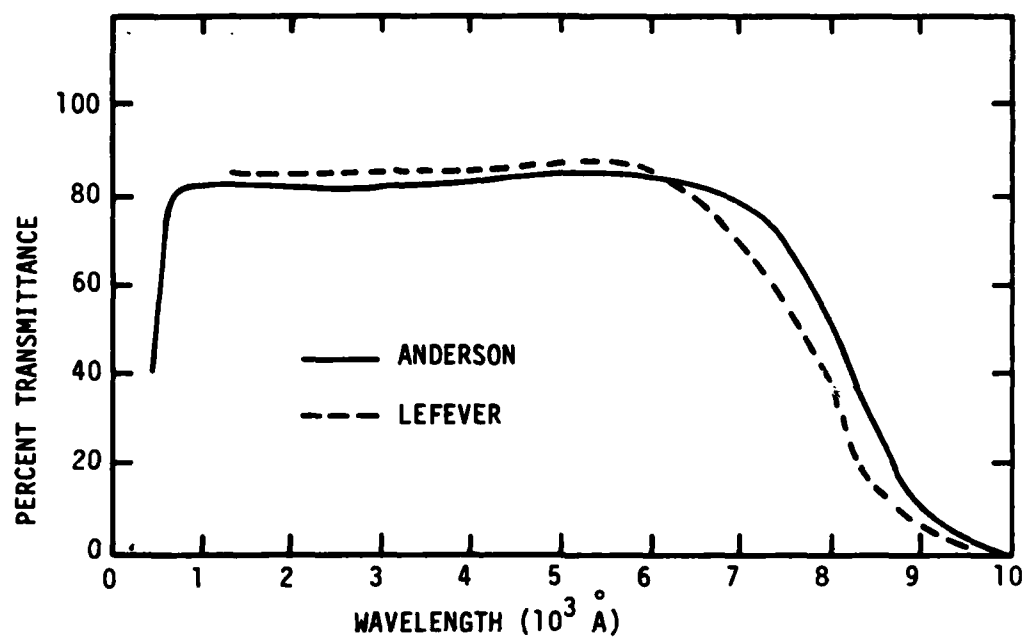
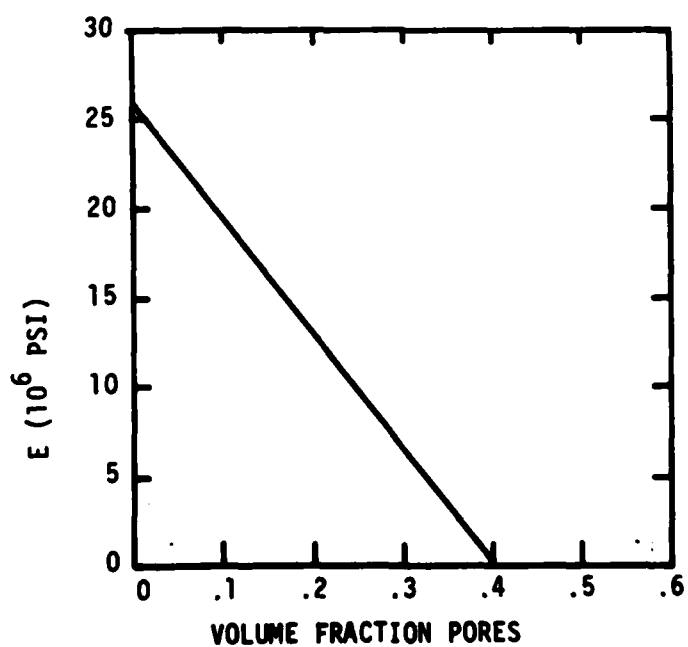Figure 1.   Transmittance of Yttrium Oxide

Figure 2.   Young's Modulus for Y_2O_3 at Room Temperature vs Volume
Fraction Pores

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Young's modulus is also found to decrease linearly with elevated temperatures (References 16, 19). In Figure 3, both the effects of porosity and temperature on Young's modulus are shown, as taken from Reference 16.
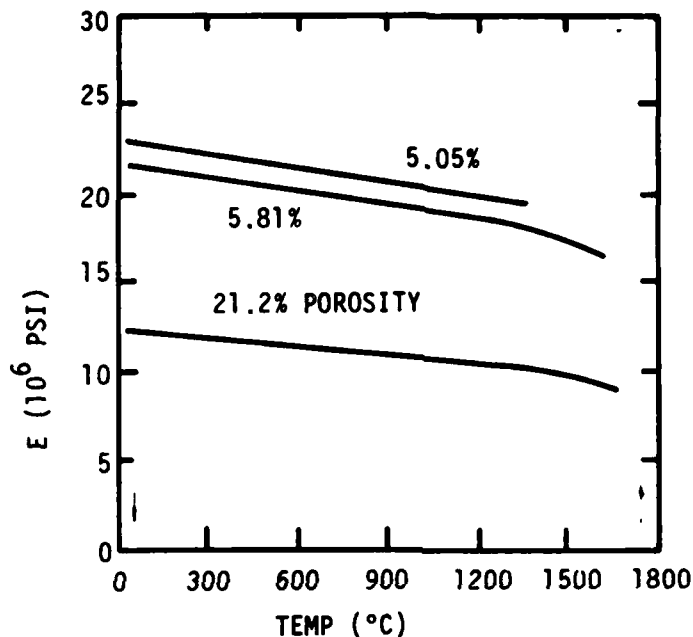

Figure 3. Young's Modulus for Y_2O_3 as a Function of Temperature for Three Levels of Porosity

Little data could be found relative to the ultimate tensile strength of yttrium oxide, particularly at elevated temperature. The modulus of rupture for Yttralox is listed as 17,000 psi at room temperature. This value is the fictitious tensile stress in the extreme fibers of a beam, computed by the flexure equation $S = \frac{Mc}{I}$ where M is the bending moment which causes rupture (Reference 20). Further insight into the high temperature behavior may be found in the Soviet literature in which the following statement was made:

"A characteristic property of Y_2O_3 is the small variation of the bending strength with an increase in the temperature right through to 1400°C, the explanation in the general case being the elastic behavior and brittle failure of the material."

Data is provided for "fine-grain dense" Y_2O_3 in which the maximum bending strength actually appears to increase by about 15% in going from room temperature to 1400°C.

Perhaps the greatest detriment to using yttrium oxide is its ability to withstand thermal shock. The combination of little plasticity at high temperature and low thermal conductivity increases the levels of thermal stress (Reference 20). A qualitative comparison is made by Anderson (Reference 11) who describes the thermal shock resistance of Yttralox as being slightly better than window glass.

To summarize this section, the properties of Yttralox are reproduced in the table below (Reference 11).

TABLE 1

PROPERTIES OF YTTRALOX CERAMIC

Density	- 5.30 gm/cc
Crystal Structure	- Cubic
Hardness	- 600 Vickers Kg/mm^2 (10 gram load)
Coefficient of Thermal Expansion	- 7.87 x 10^{-6} (Room Temp. to 900°C)
Modules of Rupture	- 17,000 psi
Modules of Elasticity	- 25,000,000 psi
Poisson's Ratio	- 0.295 at 25°C
	- 0.298 at 1000°C
Melting Point	- > 2400°C
Useful Temp. Limit in Air	- 1800°C
Porosity	- None, Fully Dense

This data is typical of that found for yttria products from several sources in the literature. Obviously, these properties must be adjusted for increased temperature porosity, etc.

3. APPLICATION TO THE RAMJET COMBUSTOR

If a ramjet combustor 6 inches in diameter and with a .25 inch thick wall is considered, the hoop stress is only 1200 psi. Thus, the yttria seems to be more than adequate in terms of strength, even when degraded for increased temperatures. The quartz tube used previously (Reference 1) was held in compression between spring loaded flanges. Thus, only a simple cylindrical shape was required. Thermal expansion during operation

was absorbed by the springs. The same configuration is proposed for
use with a yttria combustor.

Two problems remain. Thermal stress levels may be excessive. Normal
operation included fairly rapid temperature cycling when the ramjet is
ignited and extinguished. This may be relieved somewhat by extended
operation of the preheater normally used to heat the ramjet inlet air.
If the preheater is used to stabilize the ramjet combustor at an elevated
temperature before ignition and left on to gradually cool down after the
test, perhaps the thermal stress problems can be minimized.

The other problem is, of course, the actual production of a transparent
cylinder of yttrium oxide to the desired specifications. Numerous refer-
ences have been made to the ability to form objects more complicated than
simple test samples. Whether or not a transparent cylinder 6 inches in
diameter can be formed at a reasonable cost is yet to be determined. At
one time, Yttralox was advertised as available in various forms, but is
no longer produced. Thus, more research is needed in this area to deter-
mine just what the state of the art is and to locate possible sources for
manufacturing yttrium oxide components.

SECTION IV

SUMMARY AND CONCLUSIONS

Yttrium oxide appears to be a very good candidate for use in the fabrication of a transparent combustor for ramjet combustion research. Data from numerous sources shows good strength characteristics up to 1800°C. Although ramjet operating temperatures often exceed this value, a great deal of research can be accomplished at lower temperatures. If higher combustion temperatures are required, the ramjet can be extinguished before an 1800°C wall temperature is exceeded. This should still provide reasonable test times.

Whether or not a transparent yttria cylinder can be manufactured at a reasonable cost remains to be determined. Further research is needed to determine the state of the art in this area, along with direct contact with those working with the material. This report has served to compile available data on the subject with the hope of following through to the fabrication of a transparent ramjet combustor.

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